

Thin-Film Dehydration of Sirups

Eugene O. Strolle, Victor A. Turkot, and Roderick K. Eskew

Eastern Regional Research Laboratory,
Eastern Utilization Research and Development Division, Agricultural Research Service,
U. S. Department of Agriculture, Philadelphia, Pennsylvania 19118

SUMMARY

Thin-film dehydration of sirups is effected in mechanically agitated evaporators. The method is advantageous because the product leaving the evaporator is fluid enough to permit addition of natural flavors as high-fold essences. The full-flavored product is cooled, crushed, and then packed with a desiccant. Reliability of the method is evidenced by the wide variety of powders produced at this laboratory; fruit juices, berry juices, honey, and molasses have been dried by this method. Nevertheless, care must be exercised, and overdrying and underdrying avoided. Overdrying causes crystallization or increases viscosity, causing product hold-up and stalling the rotor; underdrying causes caking. Both are prevented by correctly choosing operating vacuum and product temperature. Data are presented relating these variables to product moisture, utilizing the boiling point rise concept. Also, the effect of powder composition on resistance to caking is discussed, and data are presented which will facilitate the production of free-flowing powders.

INTRODUCTION

Thin-film dehydration of fruit juices and similar materials is effected by feeding 60–80° Brix sirups to mechanically agitated thin-film vacuum evaporators, and obtaining a molten product containing 1–4% moisture. This product is then chilled on cooling rolls, ground through a 6-mesh screen, and packed with a desiccant. The production of various fruit juice products, honey, and honey-sucrose mixtures has been described by Turkot *et al.* (1956, 1960). Also, Claffey *et al.* (1961) have made a cost analysis of the dried honey process which with minor changes can be used to estimate the cost of producing any product made by this process.

The thin-film evaporator is used to evaporate heat-sensitive materials. Although high temperatures are employed, as Reay (1963) has pointed out, residence times are extremely short and heat damage is minimized. Several excellent recent reviews describe the application of this type of equipment to heat-sensitive materials (Jones, 1960; Moore and Hesler, 1963; Dedert

and Moore, 1963). However, these do not discuss the problems of dehydrating sirups in thin-film evaporators to produce practically anhydrous products. Where foodstuffs are discussed, emphasis is primarily on evaporating to sirup concentrations. These sirups are the starting materials employed in the technique which this paper discusses.

The latest review of thin-film evaporation (Najder, 1964) points out that there is an erroneous opinion that this technique is too complex to be effective and reliable. The experience at this laboratory in using this technique to produce a wide variety of dried food products gives added cogency to Najder's arguments that this technique possesses both simplicity and reliability.

Naturally, difficulties must be expected and will be encountered. However, these problems can be solved by employing a rational engineering approach. The major difficulties stem from overdrying and underdrying. If sucrose is present, overdrying leads to crystallization of the sucrose in the evaporator. This, in turn, stalls the rotor, making further operation impossible. Even though crystallization is prevented, e.g., by addition of high-maltose sirup, overdrying then causes an increase in viscosity to the point where downflow is drastically diminished, and the rotor stalls. Overdrying materials high in invert sugar or dextrose results in excessive foaming and hold-up of material. Underdrying gives trouble-free operation in the evaporator but the product cakes in packaging or during storage.

Overdrying and underdrying are prevented by the correct choice of operating conditions, i.e., vacuum and product outlet temperature. This study shows that these are the two most important variables since mass throughput was fixed and was based in this case on an evaporative load of 10–12 lb water per 1 sq ft of heating surface.

Obviously, some rational means must be available to select operating conditions that give the desired moisture content regardless of composition. The method suggested consists of relating

the boiling-point rise (BPR) to the moisture content of the product. The boiling-point rise is the difference between the product temperature and the boiling point of pure water corresponding to the vacuum obtaining during dehydration. This method in its simplest form (a dial thermometer) is used to indicate when maple sirup of desired Brix is ready to be drawn from the open-pan evaporators used in farm siruping operations (Willits, 1958). In a more sophisticated form it is used commercially in the crystallization of sucrose to indicate the degree of supersaturation of the sugar solutions (Spencer and Meade, 1945).

No data have been published relating the effect of product moisture on the boiling-point rise or showing the effect of composition on boiling-point rise corresponding to a given moisture content. This paper presents such data. Since underdrying manifests itself by caking of the product on storage, to utilize boiling-point rise data efficiently, we must also know the effects of product composition on caking and the essence composition if flavor is restored.

Restoration of natural flavor by direct incorporation of essence into the molten product leaving the evaporator is a great advantage of this process. However, depending on its own moisture content and the composition of the dried product, the added essence may raise the moisture content of the product and cause caking. If product moisture is too high, even the in-package desiccant (IPD) does not prevent caking at 72°F; storage at 38°F with IPD, however, does prevent caking. While effective in ensuring a free-flowing product, storage at 38°F is too expensive and impractical for commercial consideration.

This phenomenon may be explained as follows. The cooled solid product leaving the chilling rolls is an amorphous material that Matz (1962) termed glassy-structured. At the lower temperatures, these products retain their rigidity and brittleness. However, if the temperature is raised to the transition or softening point, the solid melts and a highly viscous supersaturated solution results. Nelson and Newton (1941), and Duck and Cross (1957) have reported that increasing the moisture content lowers the transition point and adding materials such as dextrine raises it. Duck and Cross (1957) further state that sucrose and invert sugar lower the transition point for hard candy, which is essentially a mixture of sucrose, invert sugar, and corn sirup. Turkot *et al.* (1960) reported that increasing the sucrose content raised the softening point of dried

honey. Honey is essentially a mixture of dextrose and levulose, with the latter usually in excess. Hence, these findings do not contradict those of Duck and Cross which apply to hard candy.

Once the powder has melted, crystallization may occur in the supersaturated liquid, as Heiss (1959) and Makower and Dye (1956) have pointed out. However, Palmer *et al.* (1956) reported that crystallization is not a necessary concomitant of the caking phenomenon; caking may result from the coalescence of the sticky particles after melting has occurred. Regardless of whether crystallization, coalescence, or both obtain, the product, because of the low ambient relative humidity, loses moisture, and a dry, hard, caked product results.

As pointed out above, the moisture content of full-flavored fruit juice powders comes partly from the added flavor essence. Hence, the problem of essence composition must be considered. Essences which are 700-fold or higher may be added in small amounts (1.5–2.0 ml/½ lb solid) to fruit powders such as grape and apple and still keep below caking limits defined by Turkot *et al.* (1955) for grape and apple juice products. However, it may be necessary to meet certain requirements such as the military specifications for grape powder (Military Specification, 1963), which require a minimum volatile ester content. As this paper will show, high-fold essences vary in flavor constituents, and the water content may range from 15 to 60%. Attempts to satisfy the minimum essence requirements may necessitate adding too much water, with consequent caking. In addition to the differences within the essence type, we have differences between essences, e.g., 1000-fold pineapple essence is about 90% water, which would further aggravate the caking problem.

Little information has been published on the caking of various products and the effect of added essence thereon. It is believed, however, that the studies reported here include sufficient data to choose proper sugar mixtures and operating conditions to ensure free-flowing powders.

MATERIALS AND METHODS

Dehydration. The process and equipment is shown in Fig. 1. Feed material (62–78° Brix) in which 30 ppm of food-grade silicone-base antifoaming agent has been thoroughly dispersed, is pumped from the feed tank (1) by a gear-type pump (2) through the preheater (3) to the evaporator (4). The feed pump was adjusted to deliver

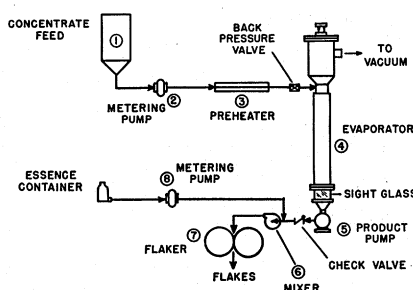


Fig. 1. Flow sheet of thin-film dehydration process.

30 lb solids/hr in all runs reported.

The evaporator is a laboratory-size Turba-Film evaporator made by Rodney Hunt Machine Company, with modifications for product removal described by Turkot *et al.* (1956, 1960). The product, which is in a molten state, is removed by product pump (5), and if flavor is to be added, this is done by a metering pump (8) which pumps 600–1000-fold essence into the molten product after it leaves the product pump. The product with its added essence enters a centrifugal mixing pump (6) and from there to the flaking rolls (7), which are cooled by 38°F water. There, the product is quickly chilled and formed into brittle flakes. These flakes are then ground through a 6-mesh screen and packed with an in-package desiccant.

Starting materials. *Grape juice products.* Grape juice product containing 33% (by weight) added sucrose was prepared by adding one volume 70° Brix sucrose solution to two volumes 70° Brix grape concentrate obtained from a commercial source. The other two grape juice products were a 72° Brix concentrate containing 25% (by weight) grape juice solids and 75% (by weight) sucrose, and a 62° Brix concentrate containing 50% (by weight) grape juice solids and 50% corn sirup solids. The corn sirup used was a 42-DE high-maltose (1435) sirup made by Corn Products Sales Division, 10 East 56th Street, New York 22, New York.

Molasses. The molasses used was an edible-grade first centrifugal molasses.

Sugar mixture. Feed material employed was a 70° Brix concentrate, the solid content of which contained 65% sucrose, 25% dextrose, and 10% corn sirup solids. The corn sirup solids was a standard-conversion 42-DE Frodex, a coarse, granular solid sirup supplied by American Maize Products Company, 250 Park Avenue, New York 17, New York. The solids content of the feed material in all cases was measured by a Abbé hand refractometer, Bausch and Lomb Optical Company,

Rochester, New York.

High-fold essences. High-fold grape and pineapple essences were prepared from 130–200-fold essences obtained from commercial sources. The essences were then concentrated in a packed column as described by Roger (1961). Feed rates ranged from 15 to 18 ml/min, and draw-off rate ranged from 1.5 to 3.0 ml/min. The high-fold essences were metered by a Mini pump into the molten product and then mixed in a centrifugal pump.

Temperature and pressure measurements. The temperatures of the product flowing into the product discharge pump, leaving the product discharge pump, and leaving the centrifugal mixing pump were measured by Fe-constantan thermocouples. These temperatures were continuously recorded by a Brown Electronik temperature recorder.

Vacuum was measured by an absolute-pressure gage, and constant vacuum was maintained by a Cartesian Manostat no. 6 made by the Emil Greiner Co., 22 Moore Street, New York City. This gage is connected by rubber tubing to the vacuum inlet at the bottom of the condenser. Steam pressure was measured by a Lonergan pressure gage with range of 0–100 psig.

Analytical procedures. *Moisture determinations.* Moisture was determined by the well known Karl Fisher method with a Serfass electric titration unit.

Volatile-esters analysis. The volatile esters were determined by the method of Walker *et al.* (1954).

Ethanol analyses. Ethanol was determined by the colorimetric method described by Reid and Truelove (1952).

Storage of products. Products in nearly all cases were packed in No. 10 cans after being ground through a 6-mesh screen. Two desiccant packages, each containing 30 g CaO, were added. The cans held 4–4.5 lb of product. All cans were stored at 72°F.

RESULTS AND DISCUSSION

Effect of starting material on operating variables. The results of the various runs are shown in Table 1. The boiling-point rises and their corresponding moisture contents for five different feed materials are shown in the last two columns. All moisture contents listed were determined on a wet basis and represent those prior to essence incorporation. Also included in Table 1 are inlet temperature, jacket steam pressure and its corresponding temperature, absolute pressure, and the boiling point of water corresponding to this pressure.

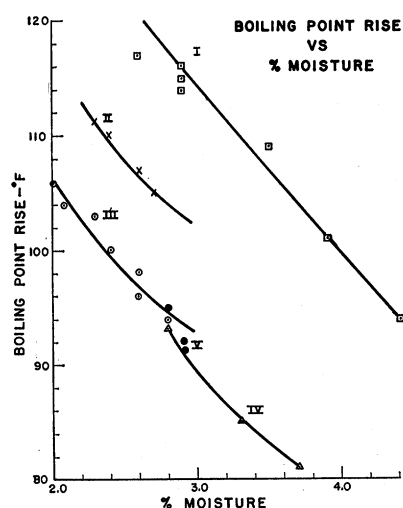


Fig. 2. Boiling-point rise vs. % moisture.

Boiling-point rise is plotted versus corresponding moisture content in Fig. 2. Curve I represents that for 50% grape juice solids and 50% high-maltose corn sirup, curve II that for 67% grape juice solids and 33% sucrose, curve III for 65% sucrose + 25% dextrose + 10% corn sirup (42 DE regular conversion), and curve IV for edible first-run molasses. The range covered for mixtures containing 25% grape juice solids + 75% sucrose was too narrow to draw a curve, but all three points listed in the table are designated on the graph by V. Curve I covers the widest range since these powders remain free-flowing over a very wide range of moistures. Curve

II covers a narrower range, and although these powders cake over 2.5% moisture at room temperature, data up to 2.7% are included for comparison. The sugar mixture represented by Curve III covers a wider range over which it is free-flowing than is included here. However, moisture content is included only up to 2.8% since under normal circumstances the higher moistures would derive in part from the added essence.

For any given material the data reveal that the higher the boiling point rise the lower is the corresponding moisture content. At this point, it should be pointed out that while pressure affects the boiling point of solutions, theoretically the product temperature alone could be used as a criterion of moisture content. However, the magnitude of change is small when compared with the corresponding magnitude of change of the boiling point of pure water. Therefore, the thermometric errors inherent in sensing and recording the product temperatures make the use of this temperature as a measure of water content highly impractical. The pressure used is that measured at the bottom of the condenser; the pressure at the product inlet would be lower due to pressure drop. Since fluid flow rate was practically identical in all cases, a constant error is introduced. Because the temperature corresponding to the pressure is used it is assumed that the magnitude of error lies within the precision limits usually required in an engineer-

ing study. To remove moisture, either the absolute pressure must be lowered by increasing vacuum or the steam pressure must be increased to raise product temperature, or else both these changes may be made. However, in any case these changes must be carried out gradually, and, furthermore, the magnitude of change is limited by practical considerations. Too much heat mars the flavor; high vacuum may cause excessive foaming, hold-up of material, or crystallization, as already described.

The effect of sucrose. Examination of Fig. 2 also reveals that a material containing higher sucrose content requires a lower boiling-point rise for a given moisture content. For example, product mixtures consisting of 75% sucrose + 25% grape juice solids require a boiling-point rise of 95°F for a 2.8% moisture content. For a 33% sucrose + 67% grape juice solids, this same moisture content (2.8%) corresponds to 103°F BPR. The three values (BPR versus % moisture) for 75% sucrose + 25% grape juice solids fall within the range of values for the sugar mixture which contains 65% sucrose, 25% dextrose and 10% corn sirup and the values for edible first-run molasses. Since first-run molasses is still fairly high in sucrose, it is to be expected that its behavior should be fairly similar to the sugar mixture and the 75% sucrose material.

The effect of high maltose sirup. Fig. 2 shows that, for a given moisture content, grape juice solids containing

Table 1. Operating variables, moisture content, and boiling-point rise for various feed materials.

Feed material	Inlet temp. (°F)	Steam pressure jacket (psig)	Steam ^a temp. jacket (°F)	Product temp. (°F)	Absolute pressure (in. Hg.)	Boiling point ^a pure water corresponding to absolute pressure (°F)	Boiling point rise (°F)	Percent ^b moisture
Grape juice	133	14.0	248	234	4.2	128	107	2.6
product	135	14-14.5	248	235	4.0	125	110	2.4
(sucrose added:	130	13.0	246	237	4.7	132	105	2.7
33 % by wt.)	138	15.0	250	236	4.0	125	111	2.3
Grape juice								
product	147	10	240	227	5.2	135	92	2.9
(sucrose added:	145	12	244	227	5.3	136	91	2.9
75 % by wt.)	145	12	244	229	5.0	134	95	2.8
Grape juice	166	20	259	240	6.8	146	94	4.4
product (corn	166	20	259	242	6.0	141	101	3.9
sirup solids,	166	20	259	240	4.6	131	109	3.5
high maltose,	165	22	263	247	4.8	133	114	2.9
42 DE added:	166	22	263	249	4.8	133	116	2.9
50 % by wt.)	166	22	263	249	4.7	132	117	2.6
	165	22	263	248	4.8	133	115	2.9
Sugar mixture	150	20	259	244	6.0	141	103	2.3
comparison: 65%	150	19	258	242	6.8	146	96	2.6
sucrose, 25%	150	20	259	243	6.7	145	98	2.6
dextrose, 10%	150	21	261	242	6.1	142	100	2.4
corn sirup	150	22	263	244	5.5	138	106	2.0
solids, regular	150	22	263	242	5.5	138	104	2.1
conversion, 42DE	150	23	264	251	9.0	157	94	2.8
Molasses, edible	150	20	259	242	10.0	162	81	3.7
first-run type	150	20	259	244	8.6	155	85	3.3
	150	22	263	250	9.0	157	93	2.8

^a From "Thermodynamic Properties of Steam," Keenan and Keyes (1936).

^b On wet basis.

Table 2. Essence retention.

Carrier	Type of essence	Dried product (g/min)	Essence (ml/min)	Volatile esters (ppm)	Volatile esters added (mg/g)	Volatile esters found in product (mg/g)	% volatile esters retained $(100 \times \frac{V.E. \text{ added}}{V.E. \text{ found}})$	Remarks
Grape juice product	Grape	227	1.70	57,000	0.43	0.412	96.5
	Grape	227	1.70	57,000	0.43	0.413	96.5	Caked ^a
	Grape	220	1.92	45,000	0.39	0.45	115
Sugar mixture	Pineapple	227	4.90	29,900	0.65	0.58	90
	Pineapple	227	4.90	29,900	0.65	0.19	29	Boiling Bubbling ^b

^a Product same lot as the one above, contained more moisture and caked during storage.

^b Product leaving discharge was boiling and bubbling.

high-maltose sirup require the highest boiling-point rise. For example, to produce products containing 2.7% moisture requires a 119°F BPR for 50% grape juice solids + 50% high maltose sirup, 105°F BPR for products containing 33% sucrose, and a 95–96°F BPR for mixtures containing 75% sucrose.

These studies have revealed the importance of feed composition in selecting operating conditions for a given moisture content. In addition, the boiling-point rise has been shown to be a reliable method for selecting these operating conditions.

Essence incorporation and the prevention of caking. *Preventing flavor loss.* Essence may be added with no significant loss of flavor constituents, as Table 2 reveals. This table includes carrier types, product and essence rates, analyses of essences and dried products, and the calculated percent volatile esters retained. Volatile esters retention values ranged from 90 to 115%; considering the trace amounts involved and inherent analytical errors, this actually means no significant loss of flavor. These data confirm the empirical organoleptic sniff test carried out by sniffing over the nip of the chilling rolls. However, there may be a considerable loss of volatile flavor, as may be seen if we look at the data for solid pineapple essence, where we have 90% and 29%, respectively. This high loss in the latter was caused by boiling and bubbling of the product as it discharges into the nip of the chilling rolls. Running the product pump and the essence-mixing pump at very high speeds may increase the temperature of the molten product as much as 18°F and cause boiling. These data show that, regardless of essence type and carrier type, essence may be incorporated efficaciously. This is important if certain requirements, e.g., those of the Armed Forces, must be met. This will now be discussed.

Essence composition and its effect on caking. In order to add a definite

amount of volatile esters it is necessary to have an analysis of the essence. This analysis and data on caking limits enable us to determine whether the requirements can be met. Table 3 shows the composition of four grape essences ranging from 700 to 1000-fold, and the composition of a 1000-fold pineapple essence. These data reveal that the latter is practically all water. Grape essences exhibit a wide variation in alcohol content (30–80%) and volatile esters which range from 10,300 to 115,000 mg/L, an almost 10-fold difference.

Because of this wide variation in water content and volatile esters, producing a powder which will meet military requirements may present difficulties. This is best illustrated by the following example. Military specifications (10 Jan 1963) require that reconstituted powdered grape beverage contain a minimum of 40 mg/L of volatile esters. This requires the powder to contain at least 0.27 mg volatile esters/g. This requirement can be met by adding 1.4 ml/min of grape essence No. 4 (Table 3). On the other hand 6.1 ml/min of essence No. 2 would be required since this contains only 10,300 ppm of volatile esters. Also, about 4.0 g of water per min would be added to the powder. Since it is impractical to dry the grape powder below 2% (because of excessive foaming and hanging-up in the evaporator) prior to adding essence, this would raise the moisture content to 3.7%. This is

above the caking limit, 2.5%. The only recourse is to use another essence or to further concentrate the essence which is low in volatile esters.

Effect of carrier composition on caking. An example will now illustrate how changing the carrier composition prevents caking. This laboratory explored the feasibility of using our thin-film dehydration process to make low-fold solid pineapple essences. This essence, when added in the ratio of two parts to one part by weight of pineapple-grapefruit solids, would have to reconstitute to a beverage containing 30 mg/L of volatile esters. This is 0.0202 mg/g solids, or, since all added flavor is to come from the sugar, 0.0606 mg/g sugar solids is required. Based on our dryer capacity of 227 g/min of product, 4.6 ml/min of high-fold pineapple essence (see Table 2) is required. If, prior to essence incorporation, the carrier moisture content is 2.0%, the additional moisture raises the moisture content to about 3.8%, and caking results. Powders consisting of 65% sucrose, 25% dextrose, and 10% corn sirup (42-DE regular conversion) at 72°F cake in the range of 3.6–3.8%; the maximum safe limit is then 3.6%.

It is possible to lower the moisture content to 1.85% prior to essence incorporation and then obtain a product containing 3.6% moisture based on adding the amount of pineapple essence given above. However, this is difficult to control, and evaporator conditions are too critical. At 1.8%

Table 3. Composition of high-fold natural fruit essences.

Number	Fruit	Fold ^a (volumetric)	Ethanol (% by wt)	Volatile esters ^b (mg/L)	Feed material	
					Fold	Volatile esters ^b (mg/L)
1	Grape	1000	80.0	115,000	150	15,000
2	Grape	1000	30.0	10,300	150	2,520
3	Grape	780	77.0	45,000	130	8,750
4	Grape	700	60.0	45,000	130	9,085
5	Pineapple	1000	10.5	29,850	200	6,990

^a Volumetric fold based on fold of essence fed to high-fold column;
 $\frac{\text{ml/min (feed)}}{\text{ml/min (overheads)}} \times \text{fold of feed}$

^b Volatile esters: measured as ethyl acetate.

Table 4. Safe upper moisture limits for storing free-flowing powders at 72°F with IPD.

Composition of powder	Safe U.L. ^a (% moisture)	Remarks
1) 67% grape juice solids 33% sucrose	2.5	Above 2.5 powder cakes
2) 25% grape juice solids 75% sucrose	3.1	Cakes 3.1-3.3
3) 50% apple juice solids 50% sucrose	2.9	Above 2.9 powder cakes
4) 12½% cranberry juice solids 87½% sucrose	3.2	Cakes at 3.3 and over
5) 50% grape juice solids 50% corn sirup (high maltose)	4.5	Highest moisture level studied Safe upper limit may be higher
6) 25% grape juice solids 37½% sucrose 37½% corn sirup (high maltose)	3.4	Highest moisture level studied Safe upper limit may be higher
7) 25% grape juice solids 56.25% sucrose 18.75% corn sirup (high maltose)	3.5	Highest moisture level studied Safe upper limit may be higher
8) 18% grape juice solids 40% sucrose 40% corn sirup (high maltose)	3.7	Highest moisture level studied Safe upper limit may be higher
9) Sugar mixture (65% sucrose 25% dextrose 10% corn sirup)	3.6	Corn sirup was 42 DE regular conversion; powders cake at 3.8%
10) Sugar mixture (65% sucrose 20% dextrose 15% corn sirup high maltose)	4.0	Highest moisture level studied Safe upper limit may be higher
11) Edible molasses (first run)	3.2	Cakes at 3.3% and over

^a All moistures reported on wet basis.

moisture and below, crystallization of the sucrose has been encountered frequently. Hence, a safe working limit of 2.0% has been adopted. Therefore, when employing an aqueous essence such as pineapple essence it is best to use a carrier containing a higher percentage of maltose to inhibit caking. A sirup composed of 65% sucrose, 20% dextrose, and 15% high-maltose corn sirup was employed. This was free-flowing even with a moisture content of 4.0%, showing how high-maltose corn sirup inhibits caking.

Some safe upper limits. Table 4 presents various mixtures and their corresponding moisture contents which may be used as safe upper limits. These data are not exhaustive, and in some cases higher upper limits may be used since a full range of moisture content in these cases was not studied. Two important points must be remembered regarding use of these data. First, the moisture contents must be considered in a relative sense since the method of determining the moisture content greatly influences the result. Second, the effect of minor constituents, e.g., acids on the transition points (and hence caking properties), has not been studied; this could be an important factor.

These data reveal that increasing the sucrose content raises the moisture level at which powder may be stored and still remain free-flowing. For example, dried grape juice products which contain 33% sucrose will cake above 2.5% moisture, while grape juice powders containing 75% sucrose will

remain free-flowing up to 3.1% moisture. The effect of sucrose seems to level off rapidly above 50% sucrose; dried apple juice product which contains 50% sucrose will cake above 2.9%, while dried cranberry products (which contain 87% sucrose) will cake above 3.2%.

The addition of high-maltose corn sirup has a more pronounced effect than sucrose in raising safe upper limits. Several examples will illustrate this. A 50:50 mixture of grape juice solids and high-maltose corn sirup did not cake even at a moisture content of 4.5%. On the other hand, a mixture of 50:50 grape juice solids and sucrose would cake between 2.5 and 3.1% moisture, probably at 3.0%, as does the apple juice product. Substituting high-maltose corn sirup for some of the sucrose in dried grape juice products raises the safe upper limit. The safe upper limit for dried products containing 25% grape juice solids and 75% sucrose is 3.1% moisture. However, mixtures containing 25% grape juice solids, 37½% sucrose, 37½% corn sirup, and mixtures containing 25% grape, 56.25% sucrose, 18.75% corn sirup did not cake at 3.5% moisture. These mixtures were not studied at a higher moisture level, so the exact upper limit is unknown. Further evidence of the effect of high-maltose corn sirup is shown by the fact that a mixture of 18% grape juice solids, 40% sucrose, and 40% corn sirup did not cake at 3.7%.

While these data on safe upper limits are far from exhaustive, they and the boiling-point rise data should

serve as convenient guideposts. Since we are dealing with heat-sensitive materials, the most important variable is product temperature. For materials highly sensitive to heat, e.g., strawberry juice, we require the lowest product temperature still giving a product easily pumped from the evaporator and yet sufficiently low in moisture to remain a free-flowing powder on storage. As has been shown, caking may be inhibited by adding sucrose or corn sirup; however, this increases the viscosity of the product leaving the evaporator, and higher temperatures must be employed to increase fluidity. With materials such as molasses, the higher temperatures required are not harmful to flavor. With materials extremely sensitive to heat, this cannot be ignored. All factors, i.e., fluidity, heat sensitivity, and resistance to caking, must be carefully considered and weighed. However, the data presented should prove helpful in solving the individual problems encountered.

REFERENCES

- Claffey, J. B., V. A. Turkot, and R. K. Eskew. 1961. Estimated cost for producing dried honey commercially. U. S. Dept. Agr., Agr. Research Service. ARS-73-33, 16 p.
- Dedert, W. G., and J. G. Moore. 1963. New trends in evaporation. *Ind. & Eng. Chem.* **55** (6), 57.
- Duck, W., and R. P. Cross. 1957. Vapor pressure study of hard candy. *Mfg. Confectioner* **37** (8), 17.
- Heiss, R. 1959. Prevention of stickiness and graining in stored hard candies. *Food Technol.* **13**, 433.
- Jones, H. H. M. 1960. Thin-layer evaporation. *Ind. Chemist* **36**, 599.
- Keenan, J. H., and F. G. Keyes. 1936. Thermodynamic properties of steam. John Wiley and Sons, New York.
- Makower, B., and W. B. Dye. 1956. Equilibrium moisture content and crystallization of amorphous sucrose and glucose. *J. Agr. Food Chem.* **4**, 72.
- Matz, S. A. 1962. Food texture. Avi Publ. Co., Westport, Connecticut, p. 153.
- Military Specification. 1963. Grape beverage, instant. MIL-G-35096, p. 2, para 3.4.2.3, 10 Jan.
- Moore, J. G., and W. E. Hesler. 1963. Evaporation of heat sensitive materials. *Chem. Eng. Progr.* **59** (2), 87.
- Najder, L. E. 1964. Thin film evaporation. *Ind. & Eng. Chem.* **56** (2), 26.
- Nelson, E. W., and R. F. Newton. 1941. The heat capacity of glucose glass. *J. Am. Chem. Soc.* **63**, 2178.
- Palmer, K. J., W. B. Dye and D. Black. 1956. X-ray diffractometer and microscopic investigation of crystallization of amorphous sucrose. *J. Agr. Food Chem.* **4**, 77.
- Reay, W. H. 1963. Recent advances in thin-film evaporation. *Ind. Chemist* **39**, 293.
- Reid, V. W., and R. K. Truelove. 1952. The colorimetric determination of alcohols. *Analyst* **77**, 325.
- Roger, N. F. 1961. The recovery of methyl anthranilate in Concord grape

- essence. *Food Technol.* **15**, 309.
- Spencer, G. L., and G. P. Meade. 1945. Cane sugar handbook. 8th ed. John Wiley & Sons, New York, p. 190.
- Turkot, V. A., H. I. Sinnamon, R. K. Eskew, G. C. W. Phillips. 1955. Storage behavior of powdered apple and grape juice products. *Food Technol.* **9**, 506.
- Turkot, V. A., R. K. Eskew, and N. C. Aceto. 1956. A continuous process for dehydrating fruit juices. *Food Technol.* **10**, 604.
- Turkot, V. A., R. K. Eskew, and J. B. Claffey. 1960. A continuous process for dehydrating honey. *Food Technol.* **14**, 387.
- Walker, L. H., G. H. Notter, R. M. McCready, and D. C. Patterson. 1954. Concentration of strawberry juice. *Food Technol.* **8**, 350.
- Willits, C. O. 1958. Maple sirup producers manual. *U. S. Dept. Agr., Agr. Handbook No. 134*, 80.
- Ms. rec'd 3/9/65.

Reference to products or companies does not imply endorsement.

The authors acknowledge with thanks the chemical analyses made by E. S. DellaMonica and his group, and the assistance of Earle Veale, of this laboratory, in carrying out the pilot-plant experiments.